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THE USE OF THE SHORT BEAM SHEAR TEST METHOD ON METAL-TO-METAL ADHESIVE BONDS

Part I, 7075-T6 Aluminum

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MECHANICS AND SURFACE INTERACTIONS BRANCH
NONMETALLIC MATERIALS DIVISION

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Final Report for period May 1979 to July 1980

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Most literature studies show that failure of an adhesive joint occurs, in general, in the region of the adhesive-oxide interface. The three major mechanical tests used today usually provide information on only the bulk adhesive. This paper presents the use of the short beam shear test as a means of loading the adhesive-oxide interface with as much shear force as possible in order to determine any subtle changes that may occur in this area during environmental testing.

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FOREWORD

This technical report was prepared by N.T. McDevitt and W. L. Baun of the Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Materials Laboratory, Air Force Wright Aeronautical Laboratories. The work was initiated under Project 2419, "Nonmetallic and Composite Materials" and WUD #44, "Improved Materials, Processes and Life Prediction Methodology of Adhesive Bonding", monitored by Dr. T. W. Haas.

This report covers work performed inhouse during the period May 1979 to July 1980.

The authors are grateful to Mr. James Solomon, Marv Knight, and Dr. N. Pagano for helpful technical discussions, and Dr. J. Whitney whose initial interest in this work provided the impetus for us to pursue this program. Mr. A. Behme is thanked for his experimental contributions.

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SECTION I

INTRODUCTION

Historically the effect of stress and environment on the overall strength of metal-to-metal adhesive bonds has been determined primarily by three mechanical tests; (1) peel, (2) wedge opening, or (3) lap shear. However, attempts to correlate data from accelerated laboratory experiments to actual service life, using these mechanical tests, have met only with marginal success (References 1-4). Actual in-service failures of an adhesive bonded structure have been reported to occur in the region of the adhesive-oxide interface. A recent study using surface instrumentation techniques (Reference 5) shows failure sites also occurring at the oxide-metal interface. Therefore, this interphase region of adhesive-oxide-metal should be of concern in laboratory studies of adhesive bond line failure. Since the joint geometry of the peel and wedge opening tests provide only crack opening data, only toughness properties of the adhesive are usually obtained and very little information is generated about the subtle changes that may occur in the interphase region. The lap shear test is capable of generating data from the interphase region; however, care must be taken on the proper choice of thickness of the test adherend in order to eliminate most of the peel forces that can occur in this test method.

When dealing with high strength aluminum alloys the aspect of corrosion has to be considered as a potential problem. At some stage in most corrosion processes a thin film is involved. The controlling factor in the

growth of this film is generally the type of environment and length of time of exposure of the test specimen. In order to study the subtle effects of surface preparation and corrosion films on the strength of the adhesive-oxide and/or oxide-metal interfaces our interest was directed to a test geometry that would be simple to fabricate and would load the bond line with as much pure shear force as possible.

We chose the short beam shear test for our study and the purpose of this paper is to illustrate the general aspects of this test method on metal-to-metal adhesive bonds. This study, to our knowledge, is the initial use of this test method on metal sandwich structures.

SECTION II

EXPERIMENTAL

1. Aluminum Alloy Specimens

Rectangular specimens were cut from a sheet of 7075-T6 bare aluminum alloy. Each specimen was 10 x 15 x 0.35 centimeters. All of the specimens were degreased with an acetone wipe, then alkaline etched with 0.1N NaOH for three minutes at room temperature. Specimens were deoxidized with a solution of 5:1 HNO_3-HF for two minutes at room temperature, then desmutted with 50% HNO_3 for thirty seconds.

Duplicate panels were then anodized in a 1.0M H_3PO_4 bath according to the conditions described in Table 1. Each pair was then bonded with FM 123-2 adhesive (in tape form), cured at 250^{o}F and 25 psi, without the aid of a primer. Specimens 3.8 x 1.3 x 0.7 centimeters were cut from the bonded panels. Duplicate specimens of each surface preparation were subjected to the environmental conditions described in Table 2.

2. Mechanical Test

The data was obtained from each specimen using an Instron test machine. The short beam shear test procedure aligns the specimen so the resultant of the applied load is perpendicular to the adhesive bond line (Figure 1). The support noses were adjusted to a span 4 times the average specimen thickness. The test recording was obtained by loading each specimen at a crosshead speed of 0.02 inch per minute until interfacial failure occurred. All tests were stopped after 6 minutes. All tests were performed at room temperature.

SECTION III

ANALYSIS OF DATA

The short beam shear test is one of the most widely used test methods for evaluation of the shearing strength of composite materials. Several assumptions are made in applying beam theory to this test, one of which is that the shear stress S is distributed across the transverse face of the specimen with a maximum at the center according to the following equation

$$S = 0.75\ (L/wt) \qquad (1)$$

where L the load applied at the center of the beam, w is the width of the beam, and t is the thickness. The fracture load (L) is obtained from the test record, and the value of S generated from the equation is assumed to represent the shearing strength of the composite.

We have utilized this same test in order to study the sandwich structure of metal-to-metal adhesive bonds. We believe this is the first attempt to apply this geometry to metal adhesive joints. We make no attempt at this time to mathematically analyze our specimen from beam theory. For this study we used the test only to determine if we could differentiate between the interfacial properties of various surface preparations that had been subjected to changing temperatures and/or environments.

Figure 2 represents the test record of a completely nonbonded specimen. This load-displacement curve was obtained from a specimen that was prepared by physically laying a film of FM 123-2 adhesive between two adherends of 7075-T6 aluminum. The initial portion of all the actual re-

corded data is nonlinear and is not displayed because it primarily represents instrument take up. The zero displacement point is obtained by dropping a tangent along the linear portion of the curve. The displacement of the specimen is plotted as a function of time. A load value, L, is obtained from the linear portion of the curve where it intercepts with the one minute mark of the abscissa. After two minutes the stiffness slope changes and the load values represent slight metal deformation mixed with the energy required to overcome the friction of the adhesive tape opposing the slide of the two opposite faces of the adherends. The load value, L_n, from this curve represents our baseline for a completely nonbonded structure.

Figure 3 represents the type of curve obtained from a specimen where the adhesive has been cured and has not been subjected to any environmental tests. This is a baseline curve for a strongly bonded specimen. Again the load value, L_b, (or slope) is obtained from the linear portion of the curve after one minute. After two minutes the slope changes and the load take up is more gradual. In this region of the bonded specimen the load values represent a slight metal deformation mixed with the energy required to overcome the adhesion of the adhesive-oxide-metal interfaces. The energy required to overcome these interfacial values is distinguished by a definitive break in the test record. The load value at this point is taken as the yield strength of the bonded interface (YSI). After the break in the curve a definite fall off in load value is recorded, a second curve will show another load value being recorded. This second portion of the record represents the load value of the deformation of the adherends plus the friction

generated by the type and degree of failure of the interface. The type of failure is distinguished by cohesive and/or adhesive. The larger the degree of adhesive failure in the interfacial region the closer the second value will parallel the curve obtained from the nonbonded specimen.

We can evaluate bond line stiffness (BLS) from the linear portion of these curves by using equation 1 and relating the data from bonded specimens to the nonbonded specimens, S_b/S_n.

$$BLS = \frac{S_b}{S_n} = \frac{L_b}{L_n} \qquad (2)$$

SECTION IV

RESULTS AND DISCUSSION

The following data represent the preliminary step in the pursuit of a testing method that will detect subtle changes in the chemistry of surfaces and interfaces when they are exposed to harsh environments. Our particular interest was to determine if changes occurring at the adhesive-oxide-metal interfaces could be detected. Figures 2 and 3 show typical test curves of a nonbonded and bonded test specimen. The bonded specimen was post cured and did not see any environmental tests. It represents a good bonded joint. Figure 4 shows a typical curve from a bonded specimen that has been subjected to environmental tests. This test record is characteristic of a degraded bond line. The YSI break in the curve is still present but at a very low value. The last type of curve recorded from this study is shown in Figure 5. This represents a bonded specimen that has totally failed interfacially due to the environment test. Although the test record is similar to the nonbonded specimen curve (Figure 2), Figure 5 does show a larger area under the curve as would be expected.

Two types of information can be obtained from the test records generated by this study. One type deals with the definitive break in the curve and we call it the yield strength of the interface (YSI). The second utilizes the initial linear portion of the curve. When the load value, obtained after one minute of displacement, is referenced to the nonbonded specimen, a bond-line-stiffness (BLS) ratio value for the cured specimen is generated.

The fact that the stress generated by the short beam shear test is driven to the interfacial region can be easily observed by a visual inspection. The determination of failure at a specific interface, adhesive-oxide or oxide-metal, can only be determined by surface instrumentation. Figure 6 shows the surfaces of specimens that were completely opened after Instron testing. These three surfaces are responsible for the test records shown in Figures 3, 4, and 5. Figure 6a is the surface of the test failed specimen considered to be a good bond. This good bond still shows a reasonable amount of adhesive failure (oxide metal surface exposed). Since a lap shear and a wedge opening test on a similarly prepared bond line showed only cohesive failure, we believe more stress is generated at the interfacial region in the short beam shear test. Figure 6b shows the surface of the low strength bond line with considerably more adhesive failure. Figure 6c shows the surface of the totally failed interface. A harsh sulfur dioxide atmosphere has generated corrosion products that are visible on these surfaces.

The test data were generated from two groups of specimens. Table 3 reports the data from the group that was cured under various conditions but were not subjected to environmental tests. Table 4 reports the data from the specimens that were subject to various temperatures and atmospheric conditions.

The data spread of the YSI values for Groups A and C is very small; therefore, the average value for these data is taken as the control value. These data are shown in Figure 7 plotted against surface preparations. The data population is small for these groups, but there is a definite indication that the 10 volt, 10 minute and 10 volt, 16 minute anodizations should provide

the more reliable surface preparation.

Data from the specimens subjected to the environmental tests are shown in Table 4. Data from the more severe tests (E, F, and G) were averaged and plotted against surface preparation (Figure 8). The data spread is larger for the environments test specimens, but this would be expected. The data population again is small; however, a definite trend toward lower values is consistent and follows the curve for the control specimens, that is the 10V, 10 minute and 10V, 16 minute surfaces give overall better results.

Before any data was obtained from the bonded specimens it was presumed that the ratio of the slopes,

$$BLS = \frac{L_b}{L_n}$$

would also give some indication as to changes in the surface preparation. Examination of the BLS values in Tables 3 and 4 does not show any trend with respect to changes in the surface. Instead the BLS values remain reasonably constant for each individual group of specimens. Therefore, the bond line stiffness value represents a figure of merit for the bulk adhesive and its response to each particular environment.

The fact that the YSI values give information on the interfacial region of the bond line and the BLS values relate primarily to bulk adhesive properties can be shown from the following analogy. Data was obtained previously (Reference 6) from a wedge opening test study using an SO_2 environment. These specimens were exposed to this environment for 168 hours at room temperature. Zero crack growth was recorded and no interfacial failures observed.

Since we know the wedge opening test generates data primarily from the bulk adhesive a zero crack growth would indicate the SO_2 environment is not very detrimental to the adhesive strength properties. At the same time we know the SO_2 environment is quite harsh for a phosphoric acid anodized aluminum alloy surface as can be seen in Figure 9 (Reference 6). More than likely the SO_2 environment has some effect on the oxide-metal interface of these (Reference 6) specimens but the stress generated at this interface by the wedge opening test was not sufficient to cause failure.

In the present study a number of specimens were subjected to this same environment (Test E and F) and then evaluated using the short beam shear test. Since the SO_2 environment is essentially selective, by having very little effect on the bulk adhesive properties but is detrimental to the oxide-metal interface, we may evaluate the BLS data as indicative of the bulk adhesive properties and the YSI data as indicative of the interfacial properties. If we use the average value of the BLS data from Test C as our control we can see there is a 1.7% change in the average BLS value of Test E. On the other hand the average value for YSI (Test E) changes by 6.4% when compared to the same value for Test C. Comparing F (the more harsh conditions) to C we can see a change of 7.5% in BLS while the YSI value changes 9.7%. It is apparent from these data that the SO_2 environment is more detrimental to the oxide-metal interface than to the bulk adhesive as was indicated by the previous study (Reference 6). However, it is also obvious as the effect of the environment becomes greater on the bond line (Test F) the two pieces of data obtained from this test cannot remain independent from each other. This is also true for the other mechanical tests, that is, when the interfacial strength becomes low enough for the mode I type crack to find it.

SECTION V

CONCLUSION

The data obtained from this preliminary study indicate the short beam shear test can be an adjunct to the mechanical tests presently being used. This test appears capable of following subtle changes that may occur in the interphase between the adhesive-oxide and oxide-metal regions of a bonded structure.

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TABLE 1. ANODIZATION CONDITIONS FOR 1.0M H_3PO_4

SURFACE PREPARATION	VOLTAGE	TIME IN BATH	AVERAGE OXIDE THICKNESS
1	10	0.5 min	225Å
2	10	2.0	1000
3	10	10.0	3200
4	10	16.0	3600
5	40	2.0	1200
6	40	5.0	3000

TABLE 2. TYPE OF EXPOSURE FOR TEST SPECIMENS

TEST	
A	specimens cured then tested within 24 hours
B	specimens cured then post cured at 220^{o} F for 8 hours
C	specimens cured then stored in **desiccator** under ambient conditions for 90 days
D	specimens from Test C subjected to dry heat, $100^{o}C$, for 63 hrs
E	specimens from Test C subjected to SO_2 environment at R.T. for 2160 hours
F	specimens from Test C subjected to SO_2 environment at $100^{o}C$ for 72 hours
G	specimens from Test C stressed to 1000 pounds for two minutes then subjected to 100% R.H. and $100^{o}C$ for 8 hours

TABLE 3. DATA FROM CONTROL SPECIMENS

SURFACE PREP	CONTROLS A		CONTROLS B		CONTROLS C	
	BLS	YSI (lbs)	BLS	YSI (lbs)	BLS	YSI (lbs)
1	1.53	2825	2.06	3050	1.84	2758
2	1.59	3012	2.15	3552	1.80	2793
3	1.55	3200	2.12	4120	1.86	3222
4	1.58	3175	2.12	4202	1.92	3202
5	1.53	3025	2.01	3725	1.84	2900
6	1.59	2875	1.98	3650	1.85	2940

TABLE 4. DATA FROM ENVIRONMENTAL TEST SPECIMENS

SURFACE PREP	ENVIRONMENT D		E		F		G	
	BLS	YSI (lbs)	BLS	YSI (lbs)	BLS	YSI (lbs)	BLS	YSI (lbs)
I	1.53	3100	1.76	2520	-	NIF*	1.62	2455
2	1.62	2960	1.82	2760	1.76	2475	1.70	2590
3	1.60	2780	1.81	2945	1.70	2820	1.73	3045
4	1.65	3210	1.82	3145	1.70	2900	1.53	2500
5	1.59	3000	1.88	2910	1.70	2515	1.56	2640
6	-	NIF*	1.82	2400	1.69	2690	1.65	2847

* No Interface Failure (See Figure 5)

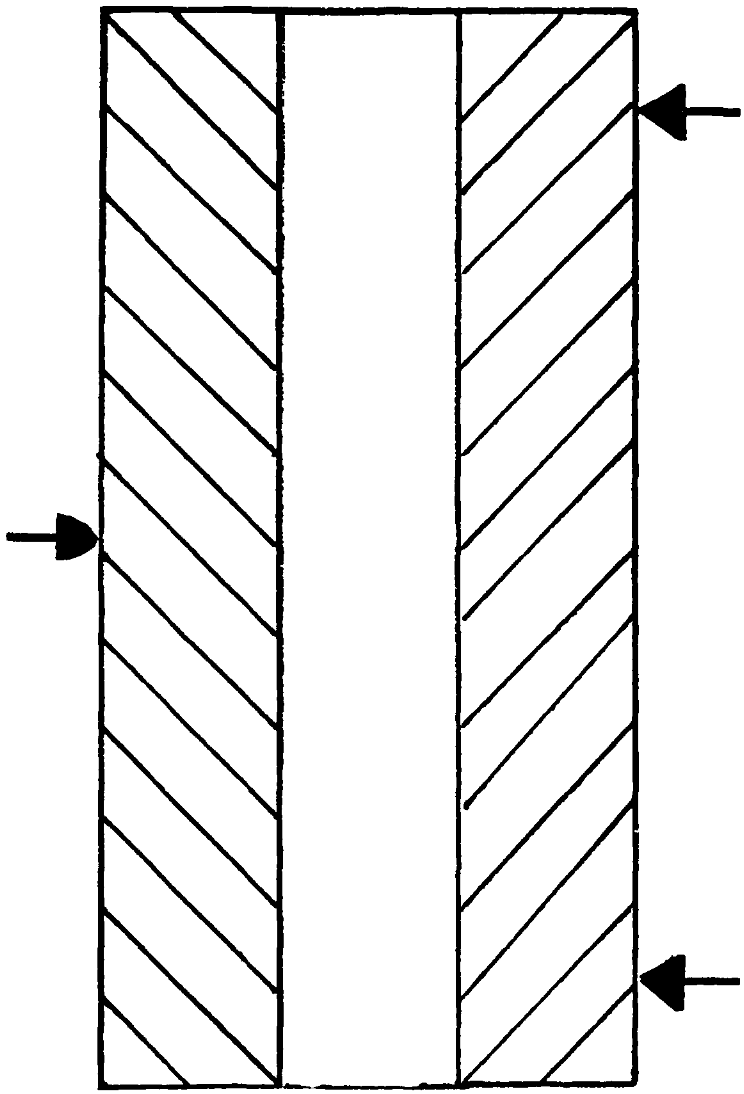

FIGURE 1. Illustration of Wedge Test Geometry

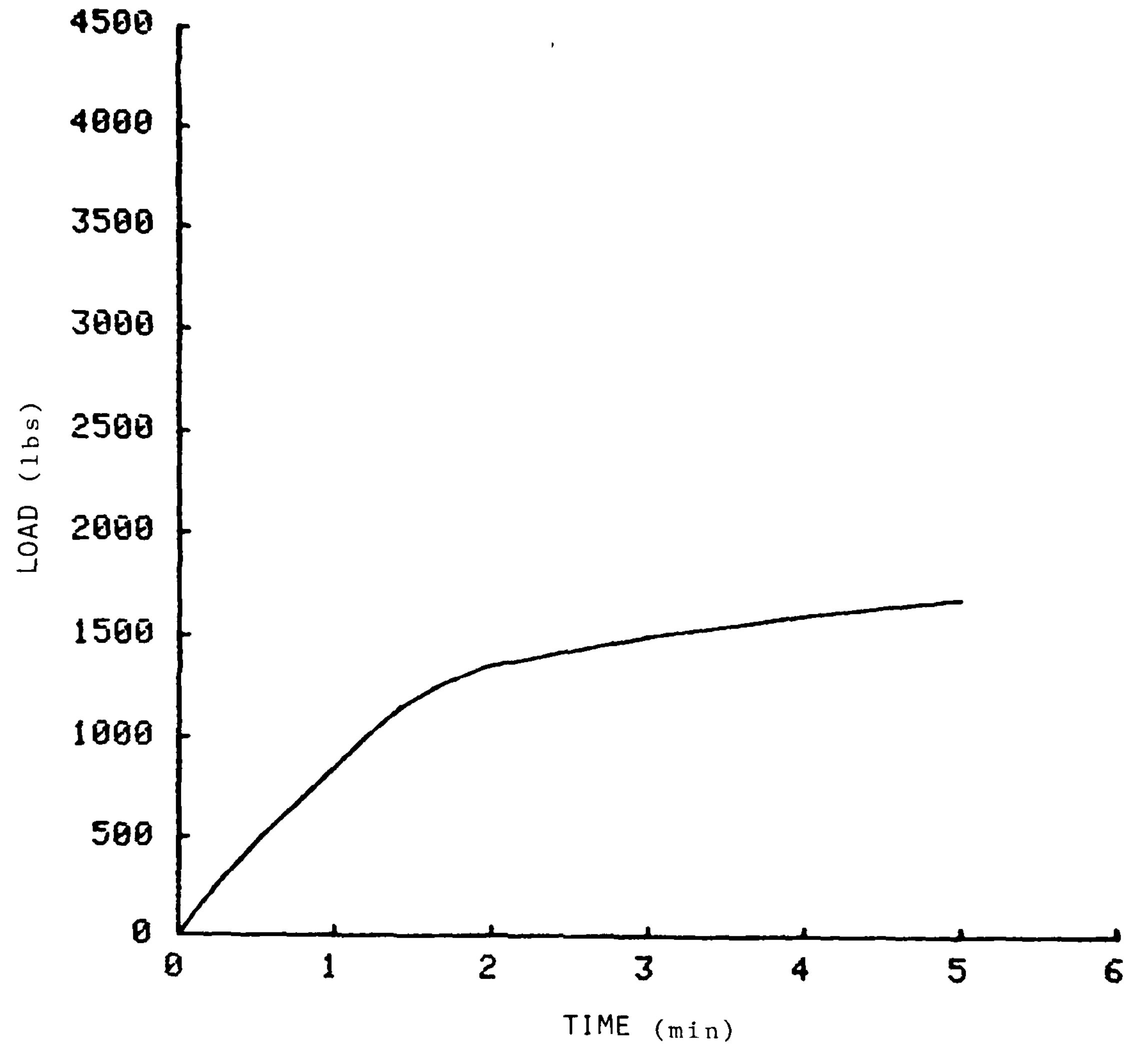

FIGURE 2. Load-Displacement Curve for Non-Bonded Specimen

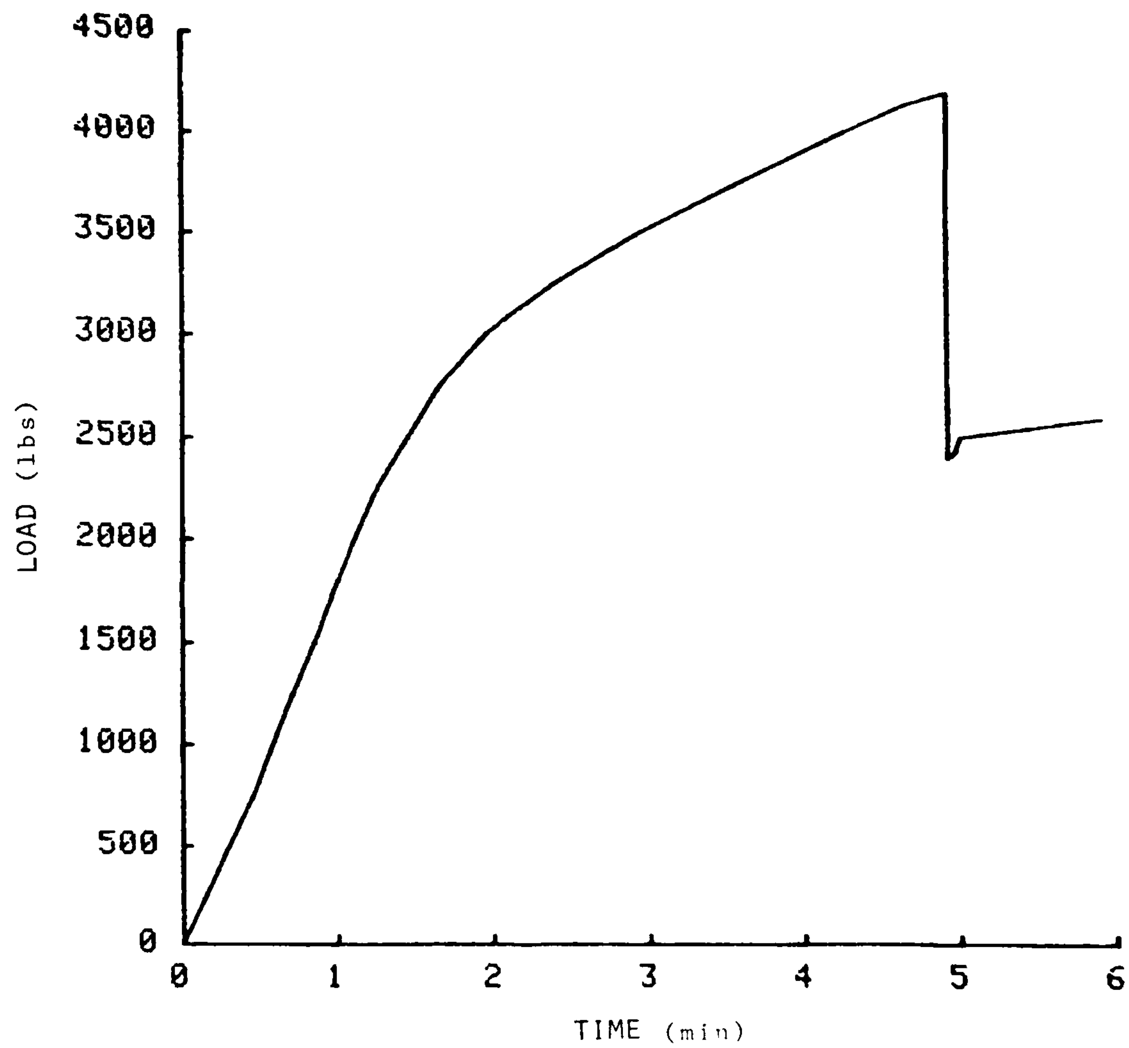

FIGURE 3. Load-Displacement Curve for Bonded and Cured Specimen

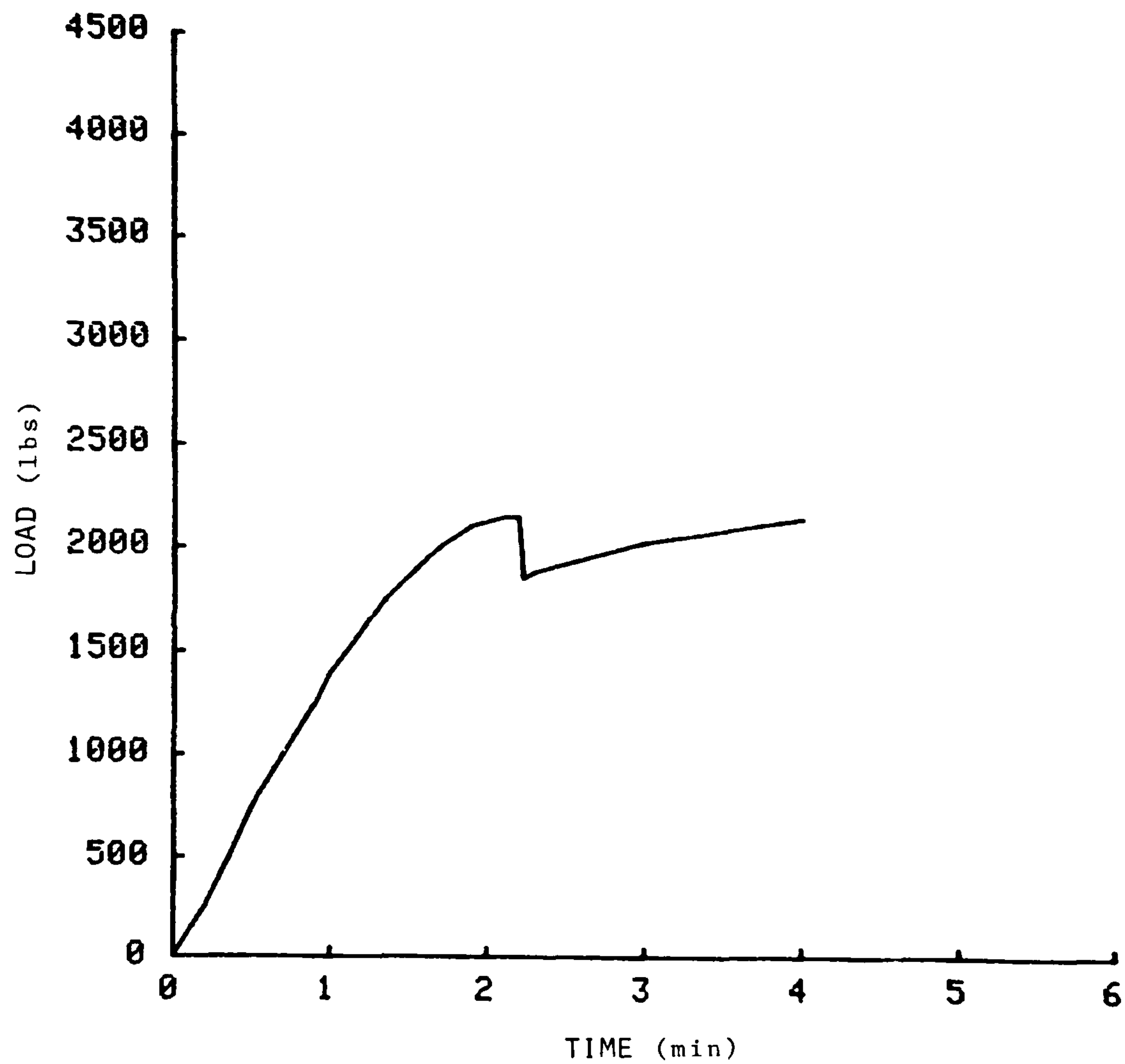

FIGURE 4. Load-Displacement Curve for Test Specimen with Low Strength Interface

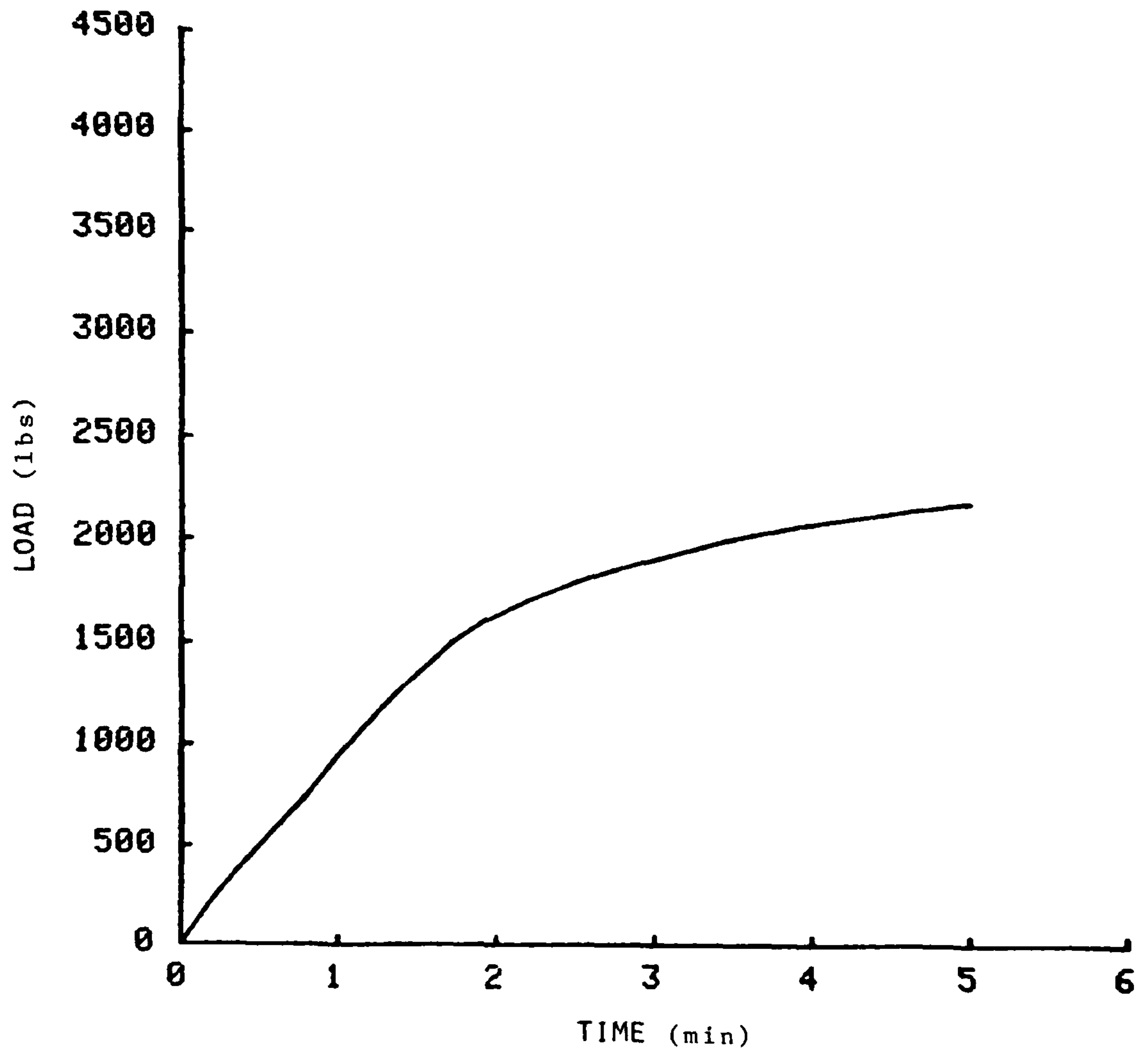

FIGURE 5. Load-Displacement Curve for Test Specimen with Adhesively Failed Interface

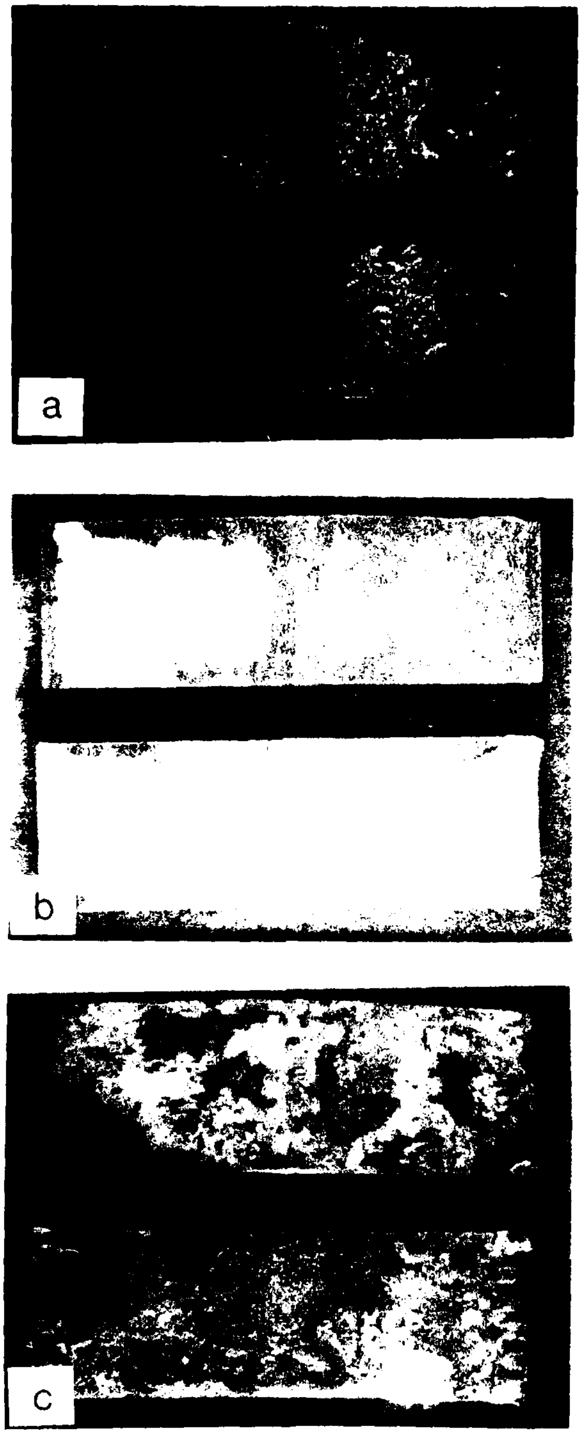

FIGURE 6. Low Magnification Photographs of Test Specimens
(a) Specimen from Figure 3, (b) Specimen from Figure 4
(c) Specimen from Figure 5

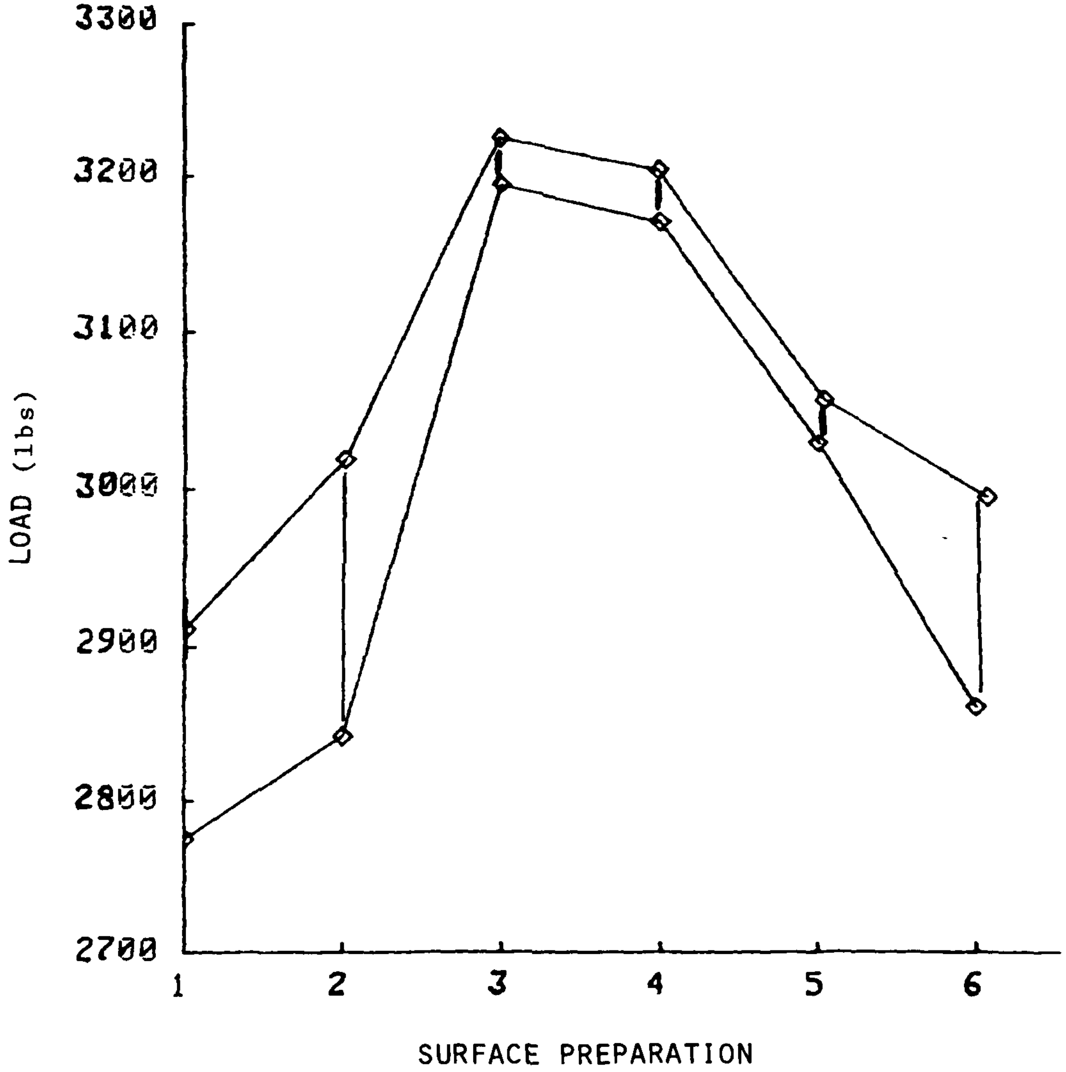

FIGURE 7. Spread of Load Values for Control Specimens

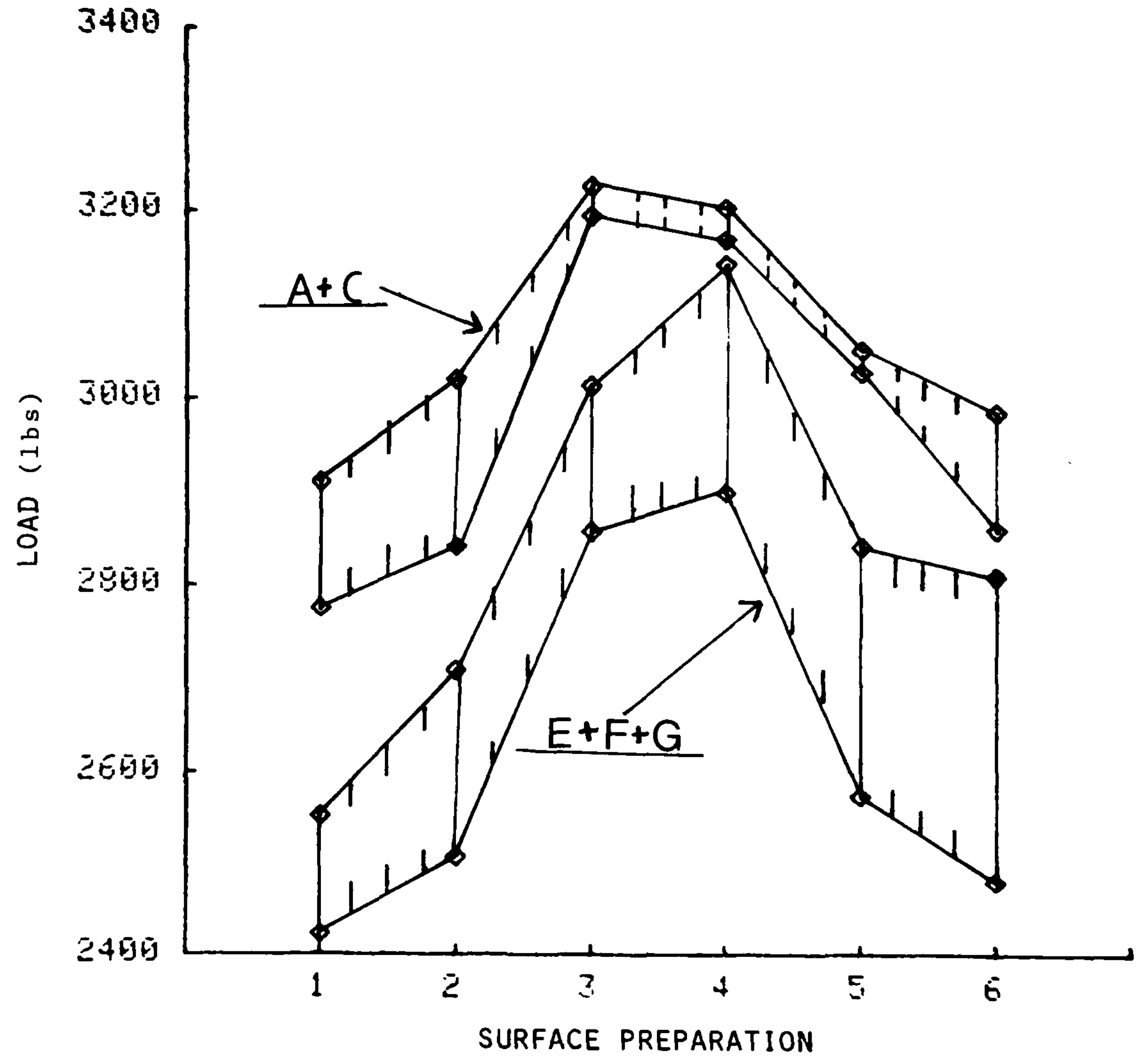

FIGURE 8. Comparison of the Spread of Load Values for Control and Test Specimens

FIGURE 9. Phosphoric Acid Anodized Surface Subjected to 30 Environment

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